



The crystal structures of six (2*E*)-3-aryl-1-(5-halogenothiophen-2-yl)prop-2-en-1-ones

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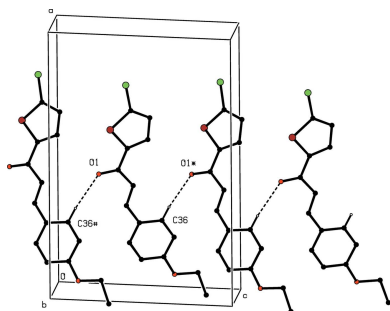
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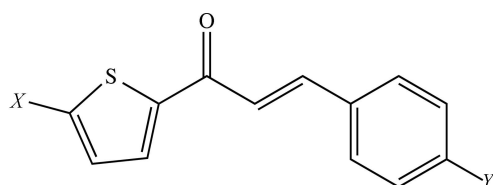
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The structures of six chalcones containing 5-halogenothiophen-2-yl substituents are reported: (2*E*)-1-(5-chlorothiophen-2-yl)-3-(4-ethylphenyl)prop-2-en-1-one, C₁₅H₁₃ClOS, (I), and (2*E*)-1-(5-bromothiophen-2-yl)-3-(4-ethylphenyl)prop-2-en-1-one, C₁₅H₁₃BrOS, (II), are isostructural in space group *P* $\bar{1}$, while (2*E*)-1-(5-chlorothiophen-2-yl)-3-(4-ethoxyphenyl)prop-2-en-1-one, C₁₅H₁₃ClO₂S, (III) and (2*E*)-1-(5-bromothiophen-2-yl)-3-(4-ethoxyphenyl)prop-2-en-1-one C₁₅H₁₃BrO₂S, (IV), are isostructural in space group *P*2₁/*c*. There are no hydrogen bonds of any kind in the structures of compounds (I) and (II), but in the structures of compounds (III) and (IV), the molecules are linked into *C*(7) chains by means of C—H...O hydrogen bonds. In the structure of (2*E*)-3-(4-bromophenyl)-1-(5-chlorothiophen-2-yl)prop-2-en-1-one, C₁₃H₈BrClOS, (V), there are again no hydrogen bonds nor π – π stacking interactions but in that of (2*E*)-1-(5-bromothiophen-2-yl)-3-(3-methoxyphenyl)prop-2-en-1-one, C₁₄H₁₁BrO₂S, (VI), the molecules are linked into *C*(5) chains by C—H...O hydrogen bonds. In each of compounds (I)–(VI), the molecular skeletons are close to planarity, and there are short halogen...halogen contacts in the structures of compounds (II) and (V) and a short Br...O contact in the structure of compound (VI). Comparisons are made with the structures of some similar compounds.

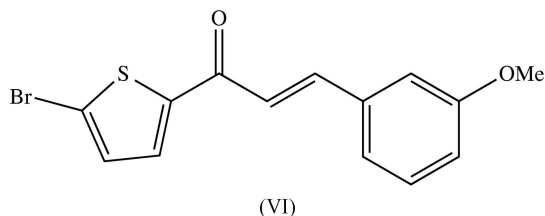
1. Chemical context

Chalcones are important constituents of many natural products, and they are abundant in edible plants where they are considered to be precursors of flavonoids and isoflavonoids. They display a wide range of pharmacological properties including antibacterial (Tang *et al.*, 2008; Kumar *et al.*, 2013*a*), anticancer (Shin *et al.*, 2013), antifungal (Domínguez *et al.*, 2001; Kumar *et al.*, 2013*a,b*), antimalarial (Li *et al.*, 1995) and antitubercular (Lin *et al.*, 2002) activity. In addition, chalcone derivatives are also important materials in photonic applications because of their excellent blue-light transmittance and good crystallization ability (Goto *et al.*, 1991; Uchida *et al.*, 1998; Indira *et al.*, 2002; Sarojini *et al.*, 2006). In a continuation of our work on chalcones containing a thiophen moiety (Naik *et al.*, 2015), six new chalcones of this type, compounds (I)–(VI) (Figs. 1–6) have now been synthesized and we report herein on their molecular structures and supramolecular assembly. Compounds (I)–(VI) were all prepared using condensation reactions, under basic conditions, between 2-acetyl-5-halogenothiophenes and substituted benzaldehydes.





- (I) $X = \text{Cl}$, $Y = \text{Et}$
 (II) $X = \text{Br}$, $Y = \text{Et}$
 (III) $X = \text{Cl}$, $Y = \text{OEt}$
 (IV) $X = \text{Br}$, $Y = \text{OEt}$
 (V) $X = \text{Cl}$, $Y = \text{Br}$



2. Structural commentary

Compounds (I) and (II) are isostructural in space group $P\bar{1}$, while compounds (III) and (IV) are isostructural in space group $P2_1/c$. Although the unit-cell dimensions for compound (V) are similar to those for compounds (I) and (II), a detailed comparison of the atomic coordinates for compounds (I), (II) and (V) indicates that there is no simple relationship between those of (I) and (II) on the one hand and those of (V) on the other. Although compound (VI) crystallizes in the same space group as compounds (III) and (IV), the unit-cell dimensions for (VI) are very different from those for (III) and (IV).

Although the molecules of compounds (I)–(VI) all lie in general positions, the non-H atoms of the molecular skeletons

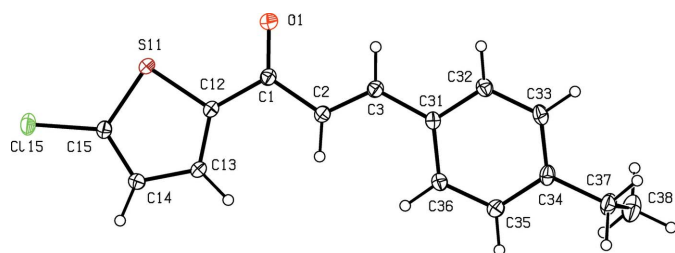


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

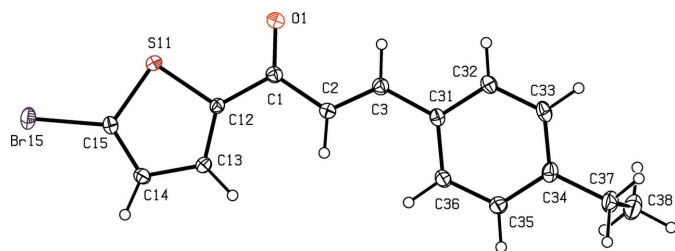


Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

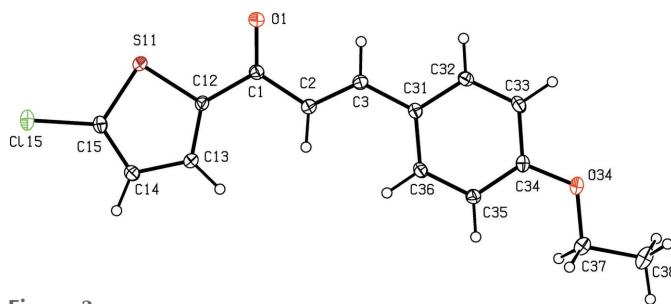


Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

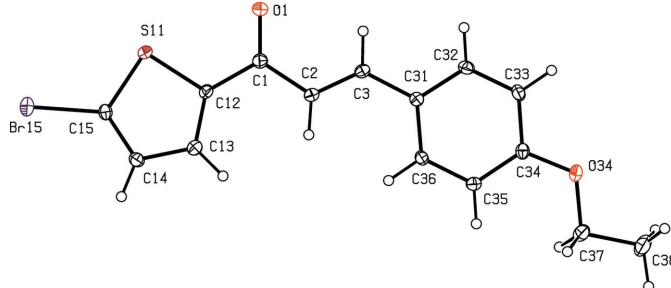


Figure 4
The molecular structure of compound (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

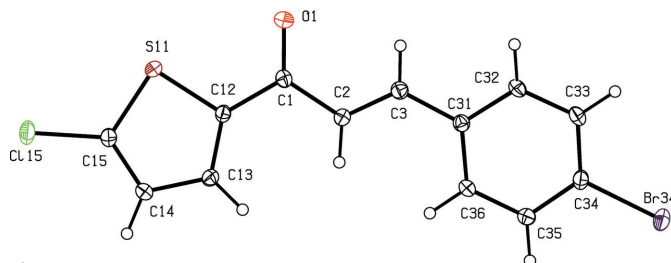


Figure 5
The molecular structure of compound (V), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

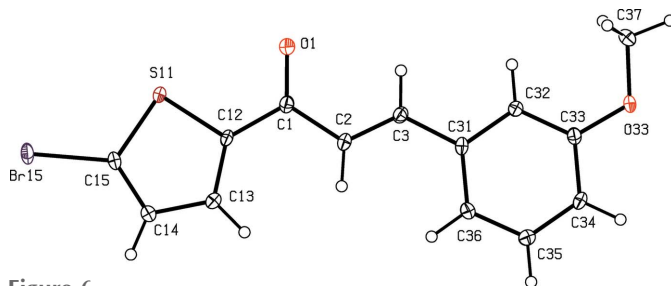


Figure 6
The molecular structure of compound (VI), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

are quite close to being co-planar, apart from the terminal C atoms of the ethyl groups in compounds (I)–(IV), as shown by the dihedral angles in Table 1. The values of these angles demonstrate the very close conformational similarity between the molecules of the three compounds, (I), (II) and (V), which crystallize in space group $P\bar{1}$, and between those of the three compounds, (III), (IV) and (VI), in $P2_1/c$. In the molecules of each of (I)–(V), the 5-halogenothiophenyl unit adopts the same orientation, with S11–C12–C1–C2 torsion angles close to 180° (Table 1). There is no evidence in any of the structures

Table 1

Selected dihedral, bond and torsion angles ($^{\circ}$) for compounds (I)–(VI).

Parameter	(I)	(II)	(III)	(IV)	(V)	(VI)
Dihedral 1	2.74 (9)	3.46 (18)	2.80 (14)	3.11 (18)	3.66 (16)	7.70 (13)
Dihedral 2	10.23 (11)	10.5 (2)	3.71 (12)	2.999(15)	9.74 (19)	3.10 (11)
Dihedral 3	11.62 (9)	11.78 (16)	6.49 (7)	6.03 (9)	11.96 (14)	5.20 (13)
C32–C33–O33						124.89 (19)
C34–C33–O33						114.96 (17)
C33–O33–C37						117.69 (16)
C33–C34–O34			116.33 (13)	116.2 (2)		
C35–C34–O34			123.98 (13)	124.2 (2)		
C34–O34–C37			117.57 (12)	117.45 (18)		
S11–C12–C1–C2	178.07 (10)	177.34 (18)	–178.36 (12)	–178.48 (16)	177.99 (19)	–172.61 (16)
C32–C33–O33–C37						7.9 (3)
C33–C34–C37–C38	–97.5 (2)	–92.6 (3)				
C33–C34–O34–C37			–175.99 (15)	–176.45 (19)		
C34–O34–C37–C38			169.60 (16)	171.3 (2)		

'Dihedral 1' represents the dihedral angle between the spacer unit (C12,C1,C2,C3,C31) and the thienyl ring. 'Dihedral 2' represents the dihedral angle between the spacer unit (C12,C1,C2,C3,C31) and the aryl ring. 'Dihedral 3' represents the dihedral angle between the thienyl and aryl rings.

reported here for orientational disorder of the type commonly observed with otherwise unsubstituted thienyl units; this is presumably a direct consequence of the presence of the halogen substituent.

In each of compounds (III), (IV) and (VI), all of which carry an alkoxy substituent, the atom C37 (Figs. 3, 4 and 6) lies close to the plane of the adjacent aryl ring: the displacements of the atoms C37 from these planes are 0.117 (3), 0.097 (4) and 0.186 (4) Å, respectively. Consistent with these observations, the corresponding pairs of exocyclic C–C–O angles (Table 1) differ significantly, as typically found for alkoxybenzenes with near-planar molecular skeletons (Seip & Seip, 1973; Ferguson *et al.*, 1996). Whereas the whole ethoxy group in each of compounds (III) and (IV) is nearly coplanar with the adjacent aryl ring, this is far from the case for compounds (I) and (II) (Table 1, Figs. 1–4).

The bond distances in compounds (I)–(VI) all lie within the usual ranges (Allen *et al.*, 1987).

3. Supramolecular interactions

There are no direction-specific intermolecular interactions in the structure of compound (I); hydrogen bonds of C–H...O and C–H... π types are absent, as are π – π stacking interactions. Hydrogen bonds and π – π stacking interactions are also absent from the structure of compound (II), but in this structure there is a short intermolecular Br...Br contact, with parameters Br15 \cdots Br15 i = 3.4917 (5) Å and C15–Br15...

Table 2

Hydrogen bond parameters (Å, $^{\circ}$) for compounds (III), (IV) and (VI).

Compound	D–H...A	D–H	H...A	D...A	D–H...A
(III)	C36–H36...O6 i	0.95	2.52	3.4649 (18)	173
(IV)	C36–H36...O6 i	0.95	2.52	3.464 (2)	172
(VI)	C13–H13...O1 ii	0.95	2.54	3.446 (3)	159

Symmetry codes: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$.

Br15 i = 151.37 (8) $^{\circ}$ [symmetry code: (i) $-x + 1, -y + 1, -z + 2$]. The Br...Br distance is significantly shorter than the van der Waals contact distance of 3.70 Å (Bondi, 1964; Rowland & Taylor, 1996), while the observed C–Br...Br angle is consistent with the results of a database analysis of such contacts (Ramasubbu *et al.*, 1986), which found that such angles were, in general, clustered around 165 $^{\circ}$.

In each of compounds (III) and (IV), a single C–H...O hydrogen bond having the carbonyl O atom as the acceptor (Table 2) links molecules related by *c*-glide symmetry into zigzag C(7) (Bernstein *et al.*, 1995) chains running parallel to the [001] direction (Fig. 7). Two chains of this type, related to one another by inversion, pass through each unit cell, but

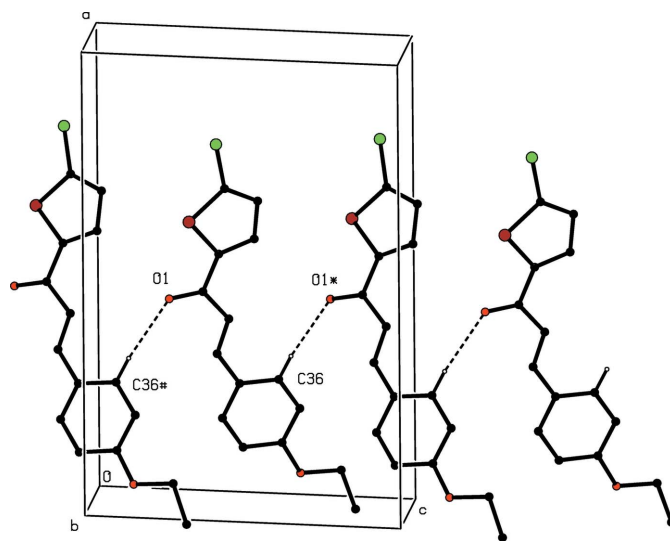


Figure 7

Part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded C(7) chain running parallel to the [001] direction. For the sake of clarity, the H atoms not involved in the motif shown have been omitted. The atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, -y + \frac{3}{2}, z + \frac{1}{2})$ and $(x, -y + \frac{3}{2}, z + \frac{1}{2})$, respectively.

there are no direction-specific interactions between adjacent chains: in particular there are no short intermolecular Br \cdots Br contacts in the structure of compound (IV), thus differing in this respect from compound (II).

There are neither hydrogen bonds nor π – π stacking interactions in the structure of compound (V). However, the structure contains a fairly short intermolecular Cl \cdots Cl contact, although, rather surprisingly, there are no short contacts of either Br \cdots Br or Br \cdots Cl types. For the contact C15–Cl15 \cdots Cl15ⁱⁱ [symmetry code: (ii) $-x + 1, -y, -z + 2$], the geometrical parameters are Cl \cdots Clⁱⁱ = 3.4825 (11) Å and C–Cl \cdots Clⁱⁱ = 167.83 (10)°. The Cl \cdots Cl distance is thus just at the van der Waals contact distance 3.48 Å (Rowland & Taylor, 1996) and so this contact cannot be regarded as structurally significant: however, it may be noted that the angle C–Cl \cdots Clⁱⁱ is entirely consistent with the results of a database analysis (Ramasubbu *et al.*, 1986).

A single C–H \cdots O hydrogen bond (Table 2) links the molecules of compound (VI) which are related by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{3}{4})$ into a $C(5)$ chain running parallel to the [010] direction (Fig. 8). Two chains of this type, related to one

another by inversion, pass through each unit cell, but there are no direction-specific interactions between adjacent chains. Not only are C–H \cdots π hydrogen bonds and π – π stacking interactions absent from the crystal structure of compound (VI), but neither are there any short Br \cdots Br contacts of the type found in compound (II). There is however a short intermolecular Br \cdots O contact with parameters Br15 \cdots O33ⁱⁱⁱ = 2.9770 (16) Å and C15–Br15 \cdots O33ⁱⁱⁱ = 167.21 (7)° [symmetry code: (iii) $x - , y, z + 1$].

All of the compounds reported here crystallize either in space group $P\bar{1}$ or in $P2_1/c$, and there appear to be some interesting connections between the space groups and the nature of the direction-specific intermolecular interactions manifested in the various structures. Thus although all six of the compounds described here contain carbonyl groups, only in compounds (III), (IV) and (VI) do the O atoms of these units participate as acceptors in C–H \cdots O hydrogen bonds: these happen to be the three examples which crystallize in space group $P2_1/c$. Of the three 5-bromothiophenyl derivatives reported here, a short Br \cdots Br contact occurs only in compound (II), the only example of this group which crystallizes in space group $P\bar{1}$.

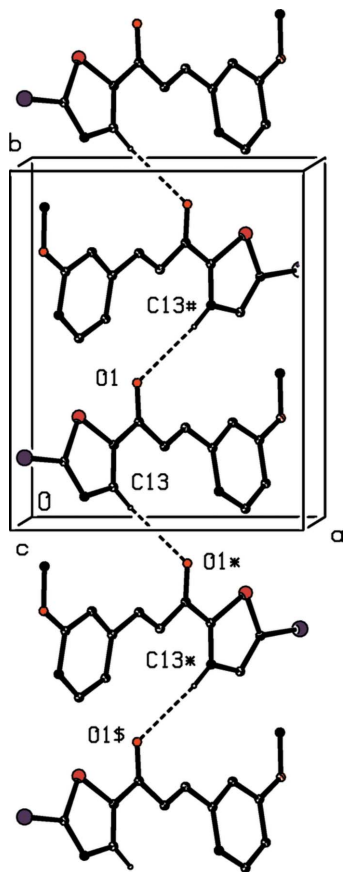


Figure 8

Part of the crystal structure of compound (VI), showing the formation of a hydrogen-bonded $C(5)$ chain running parallel to the [010] direction. For the sake of clarity, the H atoms not involved in the motif shown have been omitted. The atoms marked with an asterisk (*), a hash (#) or a dollar sign (\$) are at the symmetry positions $(-x + 1, y - \frac{1}{2}, -z + \frac{3}{2})$, $(-x + 1, y + \frac{1}{2}, -z + \frac{3}{2})$ and $(x, y - 1, z)$, respectively.

4. Database survey

The structures of a number of (2*E*)-3-aryl-1-(5-chlorothiophen-2-yl)-prop-2-en-1-one derivatives closely related to compounds (I)–(VI) have been reported recently, usually in the form of brief reports on single structures in which no comparisons with related compounds were made, and sometimes with little or no mention of the supramolecular assembly. It is thus of interest briefly to compare the supramolecular assembly in these compounds with that in compounds (I)–(VI). Compound (VII) (see Scheme below) is isomeric with compound (V), and these two compounds differ only in the exchange of the halogen location. Despite this, they are not isomorphous as compound (VII) crystallizes in space group $P2_1/c$ (Kavitha *et al.*, 2013), as opposed to $P\bar{1}$ for compound (V). There are two C–H \cdots π contacts in the structure of compound (VII), but both of these have long H \cdots D distances and small D–H \cdots A angles, and so are probably not structurally significant. There is, however, a short intermolecular Br \cdots Cl contact for which the Br \cdots Cl distance of 3.5746 (11) Å (not 3.698 (1) Å as stated in the original report), is larger than the sum, 3.55 Å (Rowland & Taylor (1996), of the van der Waals radii.

For compound (VIII) (Vepuri *et al.*, 2012), which provides a genuine example of $Z' = 2$ in space group Cc (Baur & Kassner, 1992; Marsh, 1997, 2004), there are no significant direction interactions in the structure: in particular there are neither C–H \cdots O hydrogen bonds nor short Br \cdots Br contacts. Compounds (IX) (Prabhu *et al.*, 2011*b*) and (X) (Prabhu *et al.*, 2014) are isostructural, and (X) was described as forming chains built from two independent C–H \cdots O hydrogen bonds. However, one of these contacts involves a methyl C–H bond and the other has a C–H \cdots O angle of only 130° (*cf.* Wood *et al.*, 2009), so that neither can be regarded as struc-

turally significant. On the other hand the structure of (IX) contains a significant aromatic π - π stacking interaction between the phenyl rings of inversion-related molecules, although this was apparently overlooked in the original report. The phenyl rings of the molecules at (x, y, z) and $(-x + 2, -y + 2, -z + 2)$ are strictly parallel with an interplanar spacing of 3.5113 (8) Å: the ring centroid separation is 3.6535 (11) Å, corresponding to a ring-centroid offset of 1.009 (2) Å, so leading to the formation of a centrosymmetric π -stacked dimer (Fig. 9).

The original report on compound (XI) (Sunitha *et al.*, 2012) provides no analysis or description of the supramolecular assembly. Examination of the original atomic coordinates shows firstly that molecules related by a c -glide plane are linked by a nearly linear C—H...O hydrogen bond, forming a $C(6)$ chain running parallel to the [001] direction, and secondly that inversion-related pairs of molecules are linked

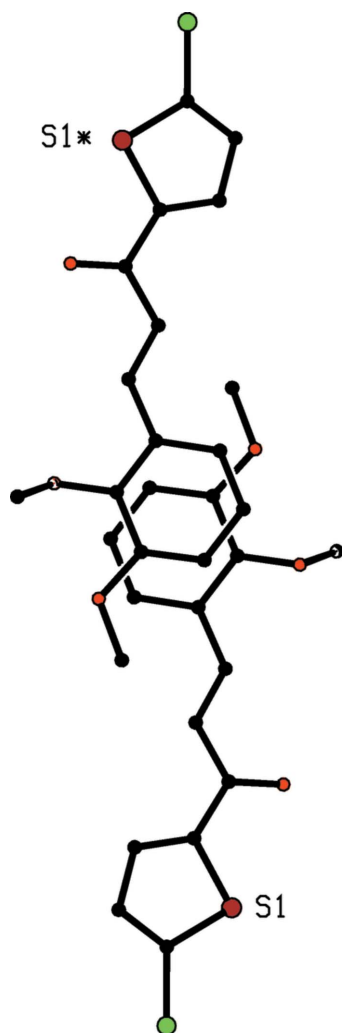
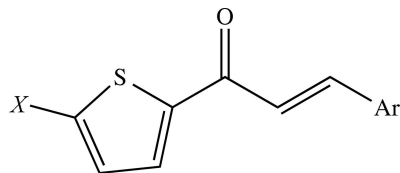


Figure 9

Part of the crystal structure of compound (IX), showing the formation of a centrosymmetric π -stacked dimer. For the sake of clarity, the H atoms and the unit-cell outline have been omitted. The original atomic coordinates (Prabhu *et al.*, 2011b) have been used and the S atom marked with an asterisk (*) is at the symmetry position $(-x + 2, -y + 2, -z + 2)$.

by a π - π stacking interaction involving the phenyl rings of the molecules at (x, y, z) and $(-x + 1, -y + 1, -z)$, with interplanar spacing 3.4465 (10) Å, ring-centroid separation 3.749 (3) Å and ring-centroid offset 1.475 (3) Å. The combined effect of these two types of interaction is the formation of a sheet lying parallel to (100); see Fig. 10.



- (VII) $X = \text{Br}$, $\text{Ar} = 4\text{-ClC}_6\text{H}_4$
 (VIII) $X = \text{Br}$, $\text{Ar} = 2\text{-BrC}_6\text{H}_4$
 (IX) $X = \text{Cl}$, $\text{Ar} = 2,3\text{-(MeO)}_2\text{C}_6\text{H}_3$
 (X) $X = \text{Br}$, $\text{Ar} = 2,3\text{-(MeO)}_2\text{C}_6\text{H}_3$
 (XI) $X = \text{Br}$, $\text{Ar} = 2,3,4\text{-(MeO)}_3\text{C}_6\text{H}_2$
 (XII) $X = \text{Cl}$, $\text{Ar} = 2,4,5\text{-(MeO)}_3\text{C}_6\text{H}_2$
 (XIII) $X = \text{Br}$, $\text{Ar} = 3,4,5\text{-(MeO)}_3\text{C}_6\text{H}_2$

There are two intermolecular C—H...O contacts in the structure of compound (XII) which were described (Prabhu *et al.*, 2011a) as joining the molecules into chains: however, for these two contacts the H...O distances, 2.68 and 2.71 Å, both exceed the sum of the van der Waals radii, 2.65 Å (Rowland & Taylor, 1996), so that these contacts certainly cannot be regarded as hydrogen bonds. Simple $C(11)$ chains are formed in the structure of compound (XIII) built from C—H...O hydrogen bonds (Vepuri *et al.*, 2011), but there are no short Br...Br contacts in either of (XI) and (XIII).

5. Synthesis and crystallization

For the synthesis of each compound, an equimolar mixture (0.01 mol of each component) of the appropriate 2-acetyl-5-halogenothiophene and the appropriately-substituted benzaldehyde was dissolved in a mixture of methanol (20 ml) and aqueous sodium hydroxide solution (5 ml of 30% w/v solu-

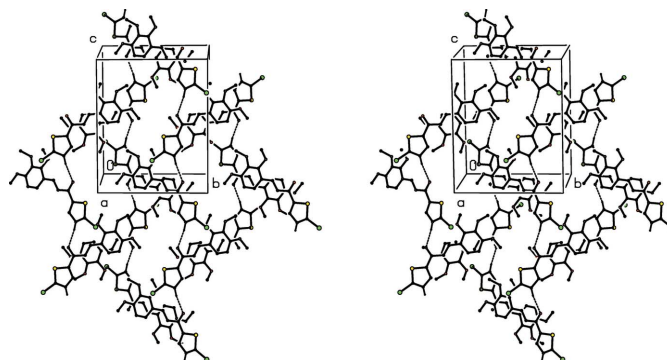


Figure 10

A stereoview of part of the crystal structure of compound (XI), showing the formation of sheets parallel to (100) built from π -stacked hydrogen-bonded $C(6)$ chains. The original atomic coordinates (Sunitha *et al.*, 2012) have been used and, for the sake of clarity, the H atoms not involved in the motif shown have been omitted.

Table 3
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	C ₁₅ H ₁₃ ClOS	C ₁₅ H ₁₃ BrOS	C ₁₅ H ₁₃ ClO ₂ S
<i>M_r</i>	276.76	321.21	292.76
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$	Triclinic, <i>P</i> $\bar{1}$	Monoclinic, <i>P</i> ₂ /c
Temperature (K)	173	173	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.0154 (5), 8.6358 (5), 14.0548 (9)	5.9745 (6), 8.6636 (7), 14.3039 (12)	16.3577 (6), 7.4518 (4), 11.0892 (4)
α , β , γ (°)	74.428 (5), 88.225 (6), 70.417 (6)	74.731 (7), 88.146 (7), 70.334 (8)	90, 92.260 (3), 90
<i>V</i> (Å ³)	661.23 (8)	671.29 (11)	1350.66 (10)
<i>Z</i>	2	2	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.43	3.20	0.43
Crystal size (mm)	0.58 × 0.32 × 0.29	0.45 × 0.22 × 0.16	0.50 × 0.28 × 0.17
Data collection			
Diffractionmeter	Agilent Eos Gemini	Agilent Eos Gemini	Agilent Eos Gemini
Absorption correction	Multi-scan (<i>CrysAlis RED</i> ; Agilent, 2012)	Multi-scan (<i>CrysAlis RED</i> ; Agilent, 2012)	Multi-scan (<i>CrysAlis RED</i> ; Agilent, 2012)
<i>T_{min}</i> , <i>T_{max}</i>	0.750, 0.883	0.326, 0.599	0.789, 0.929
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	6596, 3861, 3262	6997, 3915, 3089	7925, 3933, 3102
<i>R_{int}</i>	0.028	0.042	0.038
(sin θ/λ) _{max} (Å ⁻¹)	0.703	0.703	0.703
Refinement			
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.042, 0.119, 1.06	0.041, 0.090, 1.04	0.041, 0.111, 1.06
No. of reflections	3861	3915	3933
No. of parameters	165	165	173
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.57, -0.34	0.59, -0.63	0.28, -0.39
	(IV)	(V)	(VI)
Crystal data			
Chemical formula	C ₁₅ H ₁₃ BrO ₂ S	C ₁₃ H ₈ BrClOS	C ₁₄ H ₁₁ BrO ₂ S
<i>M_r</i>	337.21	327.60	323.19
Crystal system, space group	Monoclinic, <i>P</i> ₂ /c	Triclinic, <i>P</i> $\bar{1}$	Monoclinic, <i>P</i> ₂ /c
Temperature (K)	173	173	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	16.5498 (7), 7.5069 (4), 11.1574 (5)	6.0152 (8), 8.5691 (12), 13.1824 (9)	9.2726 (6), 11.3948 (8), 12.1472 (7)
α , β , γ (°)	90, 92.618 (4), 90	75.25 (1), 81.446 (8), 70.281 (12)	90, 93.273 (6), 90
<i>V</i> (Å ³)	1384.72 (11)	617.09 (14)	1281.37 (14)
<i>Z</i>	4	2	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	3.11	3.69	3.36
Crystal size (mm)	0.58 × 0.32 × 0.29	0.41 × 0.20 × 0.18	0.54 × 0.42 × 0.31
Data collection			
Diffractionmeter	Agilent Eos Gemini	Agilent Eos Gemini	Agilent Eos Gemini
Absorption correction	Multi-scan (<i>CrysAlis RED</i> ; Agilent, 2012)	Multi-scan (<i>CrysAlis RED</i> ; Agilent, 2012)	Multi-scan (<i>CrysAlis RED</i> ; Agilent, 2012)
<i>T_{min}</i> , <i>T_{max}</i>	0.261, 0.405	0.298, 0.514	0.216, 0.353
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	8866, 4040, 3189	6674, 3599, 2817	8260, 3722, 2914
<i>R_{int}</i>	0.037	0.026	0.035
(sin θ/λ) _{max} (Å ⁻¹)	0.703	0.703	0.703
Refinement			
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.038, 0.078, 1.03	0.042, 0.100, 1.03	0.034, 0.075, 1.02
No. of reflections	4040	3599	3722
No. of parameters	174	154	164
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.54, -0.43	1.43, -0.53	0.49, -0.46

Computer programs: *CrysAlis PRO* and *CrysAlis RED* (Agilent, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2008).

tion). The mixtures were all stirred at ambient temperature for 4 h, and then poured into ice-cold water (250 ml): the resulting solid products were collected by filtration and dried in air at 323 K. Crystals suitable for single-crystal X-ray diffraction

were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in acetone: melting points: (I) 384 K, (II) 423 K, (III) 415 K. (IV) 403 K, (V) 423 K and (VI) 390 K.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were located in difference Fourier maps and subsequently treated as riding atoms in geometrically idealized positions with C—H distances 0.95 Å (alkenyl, aromatic and heteroaromatic), 0.98 Å (CH₃) or 0.99 Å (CH₂), and with $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C})$, where $k = 1.5$ for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for other H atoms. The low-angle reflections ($\bar{1}, 2, 1$) for compound (III) and (2, 1, 2) for compound (VI), which had been attenuated by the beam stop, were omitted from the final refinements for these structures.

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supporting information

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The crystal structures of six (2*E*)-3-aryl-1-(5-halogenothiophen-2-yl)prop-2-en-1-ones

Vasant S. Naik, Hemmige S. Yathirajan, Jerry P. Jasinski, Victoria A. Smolenski and Christopher Glidewell

Computing details

For all compounds, data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* and *PLATON*.

(I) (2*E*)-1-(5-Chlorothiophen-2-yl)-3-(4-ethylphenyl)prop-2-en-1-one

Crystal data

C₁₅H₁₃ClOS

$M_r = 276.76$

Triclinic, *P*1

$a = 6.0154(5) \text{ \AA}$

$b = 8.6358(5) \text{ \AA}$

$c = 14.0548(9) \text{ \AA}$

$\alpha = 74.428(5)^\circ$

$\beta = 88.225(6)^\circ$

$\gamma = 70.417(6)^\circ$

$V = 661.23(8) \text{ \AA}^3$

$Z = 2$

$F(000) = 288$

$D_x = 1.390 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4320 reflections

$\theta = 3.4\text{--}32.6^\circ$

$\mu = 0.43 \text{ mm}^{-1}$

$T = 173 \text{ K}$

Needle, colourless

$0.58 \times 0.32 \times 0.29 \text{ mm}$

Data collection

Agilent Eos Gemini

diffractometer

Radiation source: Enhance (Mo) X-ray Source

ω scans

Absorption correction: multi-scan

(*CrysAlis RED*; Agilent, 2012)

$T_{\min} = 0.750$, $T_{\max} = 0.883$

6596 measured reflections

3861 independent reflections

3262 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.4^\circ$

$h = -7 \rightarrow 8$

$k = -11 \rightarrow 12$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.119$

$S = 1.06$

3861 reflections

165 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.1938P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.57 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.34 \text{ e \AA}^{-3}$

Extinction correction: SHELXL,
 $F_c^* = k F_c [1 + 0.001 x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.019 (5)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6919 (3)	0.25997 (18)	0.55004 (11)	0.0231 (3)
O1	0.9071 (2)	0.23082 (16)	0.54537 (9)	0.0327 (3)
C2	0.5468 (3)	0.22773 (19)	0.47997 (11)	0.0238 (3)
H2	0.3798	0.2644	0.4831	0.029*
C3	0.6465 (3)	0.14771 (18)	0.41211 (11)	0.0240 (3)
H3	0.8144	0.1055	0.4146	0.029*
S11	0.73786 (7)	0.36484 (5)	0.71337 (3)	0.02478 (12)
C12	0.5682 (3)	0.33247 (18)	0.62836 (11)	0.0213 (3)
C13	0.3333 (3)	0.38074 (19)	0.64782 (11)	0.0240 (3)
H13	0.2138	0.3719	0.6091	0.029*
C14	0.2884 (3)	0.4450 (2)	0.73162 (11)	0.0261 (3)
H14	0.1363	0.4845	0.7557	0.031*
C15	0.4915 (3)	0.44302 (19)	0.77353 (11)	0.0236 (3)
Cl15	0.51445 (8)	0.51198 (6)	0.87520 (3)	0.03470 (13)
C31	0.5211 (3)	0.11904 (18)	0.33424 (11)	0.0224 (3)
C32	0.6526 (3)	0.03119 (19)	0.26918 (11)	0.0263 (3)
H32	0.8199	−0.0166	0.2795	0.032*
C33	0.5411 (3)	0.0132 (2)	0.18973 (12)	0.0282 (3)
H33	0.6337	−0.0465	0.1464	0.034*
C34	0.2974 (3)	0.08041 (19)	0.17246 (11)	0.0264 (3)
C35	0.1656 (3)	0.1642 (2)	0.23891 (12)	0.0292 (3)
H35	−0.0019	0.2096	0.2291	0.035*
C36	0.2743 (3)	0.1824 (2)	0.31864 (12)	0.0272 (3)
H36	0.1806	0.2385	0.3632	0.033*
C37	0.1759 (4)	0.0704 (2)	0.08306 (13)	0.0344 (4)
H37A	0.0277	0.0476	0.1022	0.041*
H37B	0.2794	−0.0259	0.0594	0.041*
C38	0.1187 (4)	0.2345 (3)	−0.00031 (15)	0.0464 (5)
H38A	0.0401	0.2232	−0.0568	0.070*
H38B	0.2653	0.2563	−0.0204	0.070*
H38C	0.0138	0.3299	0.0225	0.070*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0237 (7)	0.0227 (6)	0.0224 (7)	−0.0066 (5)	−0.0010 (5)	−0.0066 (5)
O1	0.0225 (5)	0.0439 (7)	0.0342 (6)	−0.0081 (5)	0.0011 (5)	−0.0188 (5)

C2	0.0226 (7)	0.0257 (7)	0.0240 (7)	−0.0077 (6)	−0.0001 (5)	−0.0085 (6)
C3	0.0240 (7)	0.0246 (7)	0.0234 (7)	−0.0078 (6)	−0.0003 (5)	−0.0067 (5)
S11	0.02067 (19)	0.0317 (2)	0.0247 (2)	−0.00974 (15)	−0.00090 (13)	−0.01084 (15)
C12	0.0220 (7)	0.0211 (6)	0.0211 (6)	−0.0077 (5)	−0.0015 (5)	−0.0057 (5)
C13	0.0218 (7)	0.0289 (7)	0.0248 (7)	−0.0108 (6)	−0.0001 (5)	−0.0102 (6)
C14	0.0223 (7)	0.0310 (7)	0.0279 (8)	−0.0099 (6)	0.0043 (6)	−0.0122 (6)
C15	0.0272 (7)	0.0247 (6)	0.0211 (7)	−0.0104 (6)	0.0009 (5)	−0.0077 (5)
Cl15	0.0419 (2)	0.0431 (2)	0.0267 (2)	−0.01768 (19)	0.00181 (16)	−0.01764 (17)
C31	0.0260 (7)	0.0221 (6)	0.0194 (6)	−0.0084 (6)	0.0016 (5)	−0.0061 (5)
C32	0.0276 (7)	0.0250 (7)	0.0251 (7)	−0.0061 (6)	0.0031 (6)	−0.0086 (6)
C33	0.0358 (8)	0.0268 (7)	0.0261 (7)	−0.0114 (6)	0.0069 (6)	−0.0137 (6)
C34	0.0373 (9)	0.0252 (7)	0.0220 (7)	−0.0161 (6)	0.0021 (6)	−0.0080 (6)
C35	0.0271 (8)	0.0344 (8)	0.0314 (8)	−0.0129 (7)	0.0024 (6)	−0.0144 (7)
C36	0.0268 (7)	0.0322 (7)	0.0269 (7)	−0.0109 (6)	0.0052 (6)	−0.0145 (6)
C37	0.0424 (10)	0.0422 (9)	0.0284 (8)	−0.0219 (8)	0.0006 (7)	−0.0155 (7)
C38	0.0593 (13)	0.0413 (10)	0.0343 (10)	−0.0091 (9)	−0.0109 (9)	−0.0118 (8)

Geometric parameters (Å, °)

C1—O1	1.2361 (19)	C31—C36	1.401 (2)
C1—C12	1.471 (2)	C32—C33	1.390 (2)
C1—C2	1.474 (2)	C32—H32	0.9500
C2—C3	1.337 (2)	C33—C34	1.386 (2)
C2—H2	0.9500	C33—H33	0.9500
C3—C31	1.466 (2)	C34—C35	1.399 (2)
C3—H3	0.9500	C34—C37	1.509 (2)
S11—C15	1.7105 (16)	C35—C36	1.385 (2)
S11—C12	1.7300 (14)	C35—H35	0.9500
C12—C13	1.374 (2)	C36—H36	0.9500
C13—C14	1.414 (2)	C37—C38	1.519 (3)
C13—H13	0.9500	C37—H37A	0.9900
C14—C15	1.365 (2)	C37—H37B	0.9900
C14—H14	0.9500	C38—H38A	0.9800
C15—Cl15	1.7146 (15)	C38—H38B	0.9800
C31—C32	1.401 (2)	C38—H38C	0.9800
O1—C1—C12	119.59 (13)	C33—C32—H32	119.6
O1—C1—C2	123.61 (14)	C31—C32—H32	119.6
C12—C1—C2	116.79 (13)	C34—C33—C32	121.34 (14)
C3—C2—C1	121.02 (14)	C34—C33—H33	119.3
C3—C2—H2	119.5	C32—C33—H33	119.3
C1—C2—H2	119.5	C33—C34—C35	117.93 (14)
C2—C3—C31	126.11 (14)	C33—C34—C37	121.69 (15)
C2—C3—H3	116.9	C35—C34—C37	120.34 (16)
C31—C3—H3	116.9	C36—C35—C34	121.33 (16)
C15—S11—C12	90.59 (7)	C36—C35—H35	119.3
C13—C12—C1	131.06 (13)	C34—C35—H35	119.3
C13—C12—S11	111.68 (11)	C35—C36—C31	120.66 (14)

C1—C12—S11	117.26 (11)	C35—C36—H36	119.7
C12—C13—C14	112.81 (13)	C31—C36—H36	119.7
C12—C13—H13	123.6	C34—C37—C38	111.86 (14)
C14—C13—H13	123.6	C34—C37—H37A	109.2
C15—C14—C13	111.43 (14)	C38—C37—H37A	109.2
C15—C14—H14	124.3	C34—C37—H37B	109.2
C13—C14—H14	124.3	C38—C37—H37B	109.2
C14—C15—S11	113.49 (11)	H37A—C37—H37B	107.9
C14—C15—Cl15	126.35 (13)	C37—C38—H38A	109.5
S11—C15—Cl15	120.16 (9)	C37—C38—H38B	109.5
C32—C31—C36	117.93 (14)	H38A—C38—H38B	109.5
C32—C31—C3	119.01 (14)	C37—C38—H38C	109.5
C36—C31—C3	122.99 (13)	H38A—C38—H38C	109.5
C33—C32—C31	120.75 (15)	H38B—C38—H38C	109.5
O1—C1—C2—C3	6.9 (2)	C12—S11—C15—Cl15	−179.17 (9)
C12—C1—C2—C3	−173.77 (13)	C2—C3—C31—C32	−179.46 (15)
C1—C2—C3—C31	−175.39 (13)	C2—C3—C31—C36	3.6 (2)
O1—C1—C12—C13	177.79 (15)	C36—C31—C32—C33	2.2 (2)
C2—C1—C12—C13	−1.6 (2)	C3—C31—C32—C33	−174.90 (13)
O1—C1—C12—S11	−2.53 (19)	C31—C32—C33—C34	−0.3 (2)
C2—C1—C12—S11	178.07 (10)	C32—C33—C34—C35	−1.4 (2)
C15—S11—C12—C13	−0.13 (12)	C32—C33—C34—C37	176.37 (14)
C15—S11—C12—C1	−179.87 (11)	C33—C34—C35—C36	1.1 (2)
C1—C12—C13—C14	179.89 (14)	C37—C34—C35—C36	−176.71 (15)
S11—C12—C13—C14	0.20 (17)	C34—C35—C36—C31	0.9 (2)
C12—C13—C14—C15	−0.18 (19)	C32—C31—C36—C35	−2.5 (2)
C13—C14—C15—S11	0.07 (17)	C3—C31—C36—C35	174.48 (14)
C13—C14—C15—Cl15	179.21 (11)	C33—C34—C37—C38	−97.5 (2)
C12—S11—C15—C14	0.03 (12)	C35—C34—C37—C38	80.2 (2)

(II) (2*E*)-1-(5-Bromothiophen-2-yl)-3-(4-ethylphenyl)prop-2-en-1-one*Crystal data*

C₁₅H₁₃BrOS
M_r = 321.21
 Triclinic, *P*1̄
a = 5.9745 (6) Å
b = 8.6636 (7) Å
c = 14.3039 (12) Å
 α = 74.731 (7)°
 β = 88.146 (7)°
 γ = 70.334 (8)°
V = 671.29 (11) Å³

Z = 2
F(000) = 324
D_x = 1.589 Mg m^{−3}
 Mo *K*α radiation, λ = 0.71073 Å
 Cell parameters from 4444 reflections
 θ = 3.4–32.8°
 μ = 3.20 mm^{−1}
T = 173 K
 Needle, colourless
 0.45 × 0.22 × 0.16 mm

Data collection

Agilent Eos Gemini
 diffractometer
 Radiation source: Enhance (Mo) X-ray Source
 ω scans

Absorption correction: multi-scan
 (CrysAlis RED; Agilent, 2012)
T_{min} = 0.326, *T_{max}* = 0.599
 6997 measured reflections

3915 independent reflections
 3089 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\text{max}} = 30.0^\circ$, $\theta_{\text{min}} = 3.4^\circ$

$h = -6 \rightarrow 8$
 $k = -12 \rightarrow 12$
 $l = -20 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.090$
 $S = 1.04$
 3915 reflections
 165 parameters
 0 restraints
 Hydrogen site location: inferred from
 neighbouring sites

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0284P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.59 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.63 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXL,
 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0046 (13)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6905 (4)	0.2599 (3)	0.54692 (19)	0.0229 (5)
O1	0.9055 (3)	0.2306 (3)	0.54185 (14)	0.0324 (4)
C2	0.5458 (4)	0.2270 (3)	0.47838 (18)	0.0243 (5)
H2	0.3775	0.2652	0.4813	0.029*
C3	0.6430 (4)	0.1457 (3)	0.41244 (19)	0.0245 (5)
H3	0.8121	0.1024	0.4149	0.029*
S11	0.73910 (10)	0.36381 (8)	0.70722 (5)	0.02472 (15)
C12	0.5672 (4)	0.3334 (3)	0.62337 (18)	0.0201 (5)
C13	0.3321 (4)	0.3826 (3)	0.64308 (19)	0.0237 (5)
H13	0.2105	0.3752	0.6051	0.028*
C14	0.2892 (4)	0.4455 (3)	0.72571 (19)	0.0257 (5)
H14	0.1361	0.4855	0.7496	0.031*
C15	0.4928 (4)	0.4419 (3)	0.76705 (18)	0.0226 (5)
Br15	0.52162 (5)	0.51602 (4)	0.87603 (2)	0.03340 (11)
C31	0.5186 (4)	0.1155 (3)	0.33650 (18)	0.0228 (5)
C32	0.6500 (4)	0.0257 (3)	0.27336 (19)	0.0260 (5)
H32	0.8183	-0.0232	0.2839	0.031*
C33	0.5396 (5)	0.0066 (3)	0.1957 (2)	0.0287 (6)
H33	0.6335	-0.0542	0.1535	0.034*
C34	0.2949 (5)	0.0745 (3)	0.17838 (19)	0.0269 (5)
C35	0.1633 (4)	0.1596 (4)	0.2431 (2)	0.0294 (6)
H35	-0.0053	0.2054	0.2335	0.035*
C36	0.2716 (4)	0.1791 (3)	0.32083 (19)	0.0270 (5)
H36	0.1767	0.2365	0.3641	0.032*
C37	0.1733 (5)	0.0654 (4)	0.0903 (2)	0.0368 (7)

H37A	0.2707	−0.0368	0.0703	0.044*
H37B	0.0168	0.0536	0.1074	0.044*
C38	0.1372 (6)	0.2197 (4)	0.0072 (2)	0.0462 (8)
H38A	0.0542	0.2101	−0.0479	0.069*
H38B	0.2921	0.2290	−0.0119	0.069*
H38C	0.0414	0.3212	0.0268	0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0230 (11)	0.0202 (12)	0.0231 (13)	−0.0046 (10)	0.0008 (10)	−0.0051 (10)
O1	0.0224 (9)	0.0440 (12)	0.0324 (11)	−0.0077 (8)	0.0034 (8)	−0.0176 (10)
C2	0.0209 (11)	0.0284 (14)	0.0249 (13)	−0.0083 (10)	0.0031 (10)	−0.0096 (11)
C3	0.0253 (12)	0.0258 (13)	0.0232 (13)	−0.0095 (10)	0.0025 (10)	−0.0070 (11)
S11	0.0193 (3)	0.0315 (4)	0.0256 (3)	−0.0089 (3)	0.0004 (2)	−0.0111 (3)
C12	0.0205 (10)	0.0213 (12)	0.0208 (12)	−0.0090 (9)	0.0006 (9)	−0.0071 (10)
C13	0.0216 (11)	0.0272 (13)	0.0261 (13)	−0.0106 (10)	0.0011 (10)	−0.0107 (11)
C14	0.0216 (11)	0.0295 (14)	0.0293 (14)	−0.0096 (10)	0.0062 (10)	−0.0131 (12)
C15	0.0275 (11)	0.0215 (12)	0.0207 (12)	−0.0095 (10)	0.0039 (10)	−0.0073 (10)
Br15	0.04273 (18)	0.03937 (19)	0.02491 (16)	−0.01791 (13)	0.00177 (11)	−0.01472 (13)
C31	0.0266 (12)	0.0234 (13)	0.0202 (12)	−0.0100 (10)	0.0046 (10)	−0.0074 (10)
C32	0.0278 (12)	0.0226 (13)	0.0275 (14)	−0.0060 (10)	0.0044 (10)	−0.0105 (11)
C33	0.0382 (14)	0.0253 (14)	0.0280 (15)	−0.0127 (11)	0.0095 (11)	−0.0146 (12)
C34	0.0360 (13)	0.0277 (14)	0.0248 (13)	−0.0197 (11)	0.0042 (11)	−0.0088 (11)
C35	0.0262 (12)	0.0346 (15)	0.0329 (15)	−0.0138 (11)	0.0044 (11)	−0.0140 (13)
C36	0.0271 (12)	0.0309 (14)	0.0271 (14)	−0.0113 (11)	0.0070 (10)	−0.0136 (12)
C37	0.0421 (15)	0.0477 (19)	0.0337 (16)	−0.0244 (14)	0.0052 (13)	−0.0212 (15)
C38	0.0543 (19)	0.048 (2)	0.0350 (18)	−0.0118 (16)	−0.0089 (15)	−0.0155 (16)

Geometric parameters (Å, °)

C1—O1	1.226 (3)	C31—C32	1.397 (3)
C1—C12	1.464 (3)	C32—C33	1.383 (3)
C1—C2	1.468 (3)	C32—H32	0.9500
C2—C3	1.328 (3)	C33—C34	1.383 (4)
C2—H2	0.9500	C33—H33	0.9500
C3—C31	1.461 (3)	C34—C35	1.394 (3)
C3—H3	0.9500	C34—C37	1.507 (3)
S11—C15	1.705 (2)	C35—C36	1.379 (3)
S11—C12	1.729 (2)	C35—H35	0.9500
C12—C13	1.367 (3)	C36—H36	0.9500
C13—C14	1.409 (3)	C37—C38	1.494 (5)
C13—H13	0.9500	C37—H37A	0.9900
C14—C15	1.358 (3)	C37—H37B	0.9900
C14—H14	0.9500	C38—H38A	0.9800
C15—Br15	1.868 (2)	C38—H38B	0.9800
C31—C36	1.393 (3)	C38—H38C	0.9800

O1—C1—C12	119.6 (2)	C33—C32—H32	119.4
O1—C1—C2	123.1 (2)	C31—C32—H32	119.4
C12—C1—C2	117.3 (2)	C34—C33—C32	121.2 (2)
C3—C2—C1	122.0 (2)	C34—C33—H33	119.4
C3—C2—H2	119.0	C32—C33—H33	119.4
C1—C2—H2	119.0	C33—C34—C35	117.6 (2)
C2—C3—C31	127.2 (2)	C33—C34—C37	121.8 (2)
C2—C3—H3	116.4	C35—C34—C37	120.5 (2)
C31—C3—H3	116.4	C36—C35—C34	121.7 (2)
C15—S11—C12	90.81 (11)	C36—C35—H35	119.2
C13—C12—C1	131.3 (2)	C34—C35—H35	119.2
C13—C12—S11	111.33 (17)	C35—C36—C31	120.7 (2)
C1—C12—S11	117.35 (16)	C35—C36—H36	119.6
C12—C13—C14	112.9 (2)	C31—C36—H36	119.6
C12—C13—H13	123.6	C38—C37—C34	112.1 (2)
C14—C13—H13	123.6	C38—C37—H37A	109.2
C15—C14—C13	111.9 (2)	C34—C37—H37A	109.2
C15—C14—H14	124.1	C38—C37—H37B	109.2
C13—C14—H14	124.1	C34—C37—H37B	109.2
C14—C15—S11	113.11 (17)	H37A—C37—H37B	107.9
C14—C15—Br15	126.95 (18)	C37—C38—H38A	109.5
S11—C15—Br15	119.93 (13)	C37—C38—H38B	109.5
C36—C31—C32	117.6 (2)	H38A—C38—H38B	109.5
C36—C31—C3	122.8 (2)	C37—C38—H38C	109.5
C32—C31—C3	119.5 (2)	H38A—C38—H38C	109.5
C33—C32—C31	121.2 (2)	H38B—C38—H38C	109.5
O1—C1—C2—C3	7.6 (4)	C12—S11—C15—Br15	−178.81 (16)
C12—C1—C2—C3	−173.3 (3)	C2—C3—C31—C36	3.5 (4)
C1—C2—C3—C31	−175.6 (2)	C2—C3—C31—C32	−179.5 (3)
O1—C1—C12—C13	177.6 (3)	C36—C31—C32—C33	2.6 (4)
C2—C1—C12—C13	−1.5 (4)	C3—C31—C32—C33	−174.6 (2)
O1—C1—C12—S11	−3.5 (3)	C31—C32—C33—C34	−0.6 (4)
C2—C1—C12—S11	177.34 (18)	C32—C33—C34—C35	−1.3 (4)
C15—S11—C12—C13	0.0 (2)	C32—C33—C34—C37	175.7 (2)
C15—S11—C12—C1	−179.1 (2)	C33—C34—C35—C36	1.2 (4)
C1—C12—C13—C14	179.0 (3)	C37—C34—C35—C36	−175.8 (3)
S11—C12—C13—C14	0.1 (3)	C34—C35—C36—C31	0.8 (4)
C12—C13—C14—C15	−0.2 (3)	C32—C31—C36—C35	−2.7 (4)
C13—C14—C15—S11	0.2 (3)	C3—C31—C36—C35	174.4 (2)
C13—C14—C15—Br15	178.79 (19)	C33—C34—C37—C38	−92.6 (3)
C12—S11—C15—C14	−0.1 (2)	C35—C34—C37—C38	84.4 (3)

(III) (2E)-1-(5-Chlorothiophen-2-yl)-3-(4-ethoxyphenyl)prop-2-en-1-one

*Crystal data*C₁₅H₁₃ClO₂S $M_r = 292.76$ Monoclinic, $P2_1/c$ $a = 16.3577$ (6) Å $b = 7.4518$ (4) Å $c = 11.0892$ (4) Å $\beta = 92.260$ (3)° $V = 1350.66$ (10) Å³ $Z = 4$ $F(000) = 608$ $D_x = 1.440$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4472 reflections

 $\theta = 3.6$ – 32.8° $\mu = 0.43$ mm⁻¹ $T = 173$ K

Needle, colourless

 $0.50 \times 0.28 \times 0.17$ mm*Data collection*

Agilent Eos Gemini

diffractometer

Radiation source: Enhance (Mo) X-ray Source

 ω scans

Absorption correction: multi-scan

(CrysAlis RED; Agilent, 2012)

 $T_{\min} = 0.789$, $T_{\max} = 0.929$

7925 measured reflections

3933 independent reflections

3102 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.038$ $\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.6^\circ$ $h = -23 \rightarrow 12$ $k = -9 \rightarrow 10$ $l = -15 \rightarrow 15$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.041$ $wR(F^2) = 0.111$ $S = 1.06$

3933 reflections

173 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0511P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.28$ e Å⁻³ $\Delta\rho_{\min} = -0.39$ e Å⁻³*Special details*

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.48158 (9)	0.8068 (2)	0.36984 (12)	0.0216 (3)
O1	0.46622 (7)	0.8563 (2)	0.26556 (9)	0.0323 (3)
C2	0.42015 (9)	0.7307 (2)	0.44768 (12)	0.0224 (3)
H2	0.4360	0.6950	0.5275	0.027*
C3	0.34250 (9)	0.7104 (2)	0.40898 (12)	0.0203 (3)
H3	0.3296	0.7470	0.3284	0.024*
S11	0.63794 (2)	0.91918 (6)	0.33294 (3)	0.02406 (12)
C12	0.56528 (9)	0.8246 (2)	0.42167 (12)	0.0200 (3)
C13	0.59796 (9)	0.7742 (2)	0.53208 (13)	0.0219 (3)
H313	0.5670	0.7205	0.5932	0.026*

C14	0.68245 (9)	0.8104 (2)	0.54546 (13)	0.0233 (3)
H14	0.7151	0.7840	0.6160	0.028*
C15	0.71118 (9)	0.8880 (2)	0.44464 (13)	0.0217 (3)
Cl15	0.81004 (2)	0.95089 (6)	0.42162 (4)	0.03018 (12)
C31	0.27552 (9)	0.6388 (2)	0.47614 (12)	0.0189 (3)
C32	0.19780 (9)	0.6176 (2)	0.41992 (13)	0.0229 (3)
H32	0.1900	0.6488	0.3372	0.027*
C33	0.13266 (9)	0.5529 (2)	0.48145 (14)	0.0245 (3)
H33	0.0807	0.5394	0.4412	0.029*
C34	0.14307 (9)	0.5074 (2)	0.60284 (13)	0.0215 (3)
C35	0.21977 (9)	0.5257 (2)	0.66077 (13)	0.0220 (3)
H35	0.2274	0.4940	0.7434	0.026*
C36	0.28472 (9)	0.5902 (2)	0.59763 (13)	0.0212 (3)
H36	0.3369	0.6017	0.6377	0.025*
O34	0.07523 (7)	0.44649 (17)	0.65798 (10)	0.0274 (3)
C37	0.08278 (10)	0.4097 (3)	0.78481 (14)	0.0304 (4)
H37A	0.1087	0.5125	0.8281	0.036*
H37B	0.1170	0.3019	0.7999	0.036*
C38	−0.00225 (12)	0.3790 (3)	0.82785 (18)	0.0427 (5)
H38A	−0.0281	0.2806	0.7817	0.064*
H38B	−0.0347	0.4886	0.8161	0.064*
H38C	0.0007	0.3476	0.9137	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0198 (7)	0.0260 (8)	0.0190 (6)	0.0008 (7)	0.0008 (5)	−0.0012 (6)
O1	0.0234 (6)	0.0528 (9)	0.0206 (5)	−0.0050 (6)	−0.0009 (4)	0.0065 (5)
C2	0.0215 (7)	0.0261 (8)	0.0196 (6)	−0.0009 (7)	0.0016 (5)	0.0003 (6)
C3	0.0214 (7)	0.0216 (8)	0.0180 (6)	0.0004 (6)	0.0018 (5)	−0.0024 (5)
S11	0.02107 (19)	0.0335 (2)	0.01778 (18)	−0.00399 (17)	0.00259 (14)	0.00242 (15)
C12	0.0185 (7)	0.0230 (8)	0.0185 (6)	−0.0016 (6)	0.0033 (5)	−0.0008 (5)
C13	0.0212 (7)	0.0255 (8)	0.0192 (6)	−0.0018 (7)	0.0026 (5)	0.0020 (6)
C14	0.0216 (7)	0.0250 (8)	0.0231 (7)	−0.0011 (7)	−0.0022 (5)	0.0013 (6)
C15	0.0185 (7)	0.0209 (8)	0.0258 (7)	−0.0007 (6)	0.0014 (6)	−0.0041 (6)
Cl15	0.01899 (19)	0.0340 (3)	0.0378 (2)	−0.00428 (17)	0.00436 (16)	−0.00219 (17)
C31	0.0175 (6)	0.0190 (8)	0.0202 (6)	0.0008 (6)	0.0009 (5)	−0.0031 (6)
C32	0.0214 (7)	0.0276 (9)	0.0195 (7)	0.0005 (7)	−0.0018 (5)	−0.0001 (6)
C33	0.0176 (7)	0.0297 (9)	0.0260 (7)	−0.0006 (7)	−0.0040 (6)	−0.0026 (6)
C34	0.0172 (7)	0.0217 (8)	0.0257 (7)	−0.0007 (6)	0.0032 (6)	−0.0024 (6)
C35	0.0202 (7)	0.0269 (8)	0.0188 (7)	0.0010 (7)	0.0008 (5)	−0.0003 (6)
C36	0.0174 (7)	0.0247 (8)	0.0213 (7)	−0.0003 (6)	−0.0021 (5)	−0.0021 (6)
O34	0.0190 (5)	0.0351 (7)	0.0283 (6)	−0.0048 (5)	0.0039 (4)	0.0023 (5)
C37	0.0279 (8)	0.0358 (10)	0.0279 (8)	−0.0025 (8)	0.0072 (7)	0.0027 (7)
C38	0.0357 (10)	0.0494 (13)	0.0440 (10)	−0.0071 (10)	0.0159 (8)	0.0049 (9)

Geometric parameters (Å, °)

C1—O1	1.2302 (17)	C32—C33	1.375 (2)
C1—C2	1.465 (2)	C32—H32	0.9500
C1—C12	1.4698 (19)	C33—C34	1.392 (2)
C2—C3	1.333 (2)	C33—H33	0.9500
C2—H2	0.9500	C34—O34	1.3654 (19)
C3—C31	1.451 (2)	C34—C35	1.394 (2)
C3—H3	0.9500	C35—C36	1.381 (2)
S11—C15	1.7049 (15)	C35—H35	0.9500
S11—C12	1.7233 (15)	C36—H36	0.9500
C12—C13	1.369 (2)	O34—C37	1.4335 (19)
C13—C14	1.410 (2)	C37—C38	1.506 (2)
C13—H313	0.9500	C37—H37A	0.9900
C14—C15	1.359 (2)	C37—H37B	0.9900
C14—H14	0.9500	C38—H38A	0.9800
C15—C115	1.7125 (15)	C38—H38B	0.9800
C31—C36	1.398 (2)	C38—H38C	0.9800
C31—C32	1.4027 (19)		
O1—C1—C2	123.41 (13)	C31—C32—H32	119.2
O1—C1—C12	119.55 (13)	C32—C33—C34	119.86 (14)
C2—C1—C12	117.04 (12)	C32—C33—H33	120.1
C3—C2—C1	121.42 (13)	C34—C33—H33	120.1
C3—C2—H2	119.3	O34—C34—C33	116.33 (13)
C1—C2—H2	119.3	O34—C34—C35	123.98 (13)
C2—C3—C31	127.28 (13)	C33—C34—C35	119.69 (14)
C2—C3—H3	116.4	C36—C35—C34	119.79 (14)
C31—C3—H3	116.4	C36—C35—H35	120.1
C15—S11—C12	90.58 (7)	C34—C35—H35	120.1
C13—C12—C1	130.54 (14)	C35—C36—C31	121.54 (14)
C13—C12—S11	111.66 (11)	C35—C36—H36	119.2
C1—C12—S11	117.79 (10)	C31—C36—H36	119.2
C12—C13—C14	112.83 (13)	C34—O34—C37	117.57 (12)
C12—C13—H313	123.6	O34—C37—C38	107.17 (14)
C14—C13—H313	123.6	O34—C37—H37A	110.3
C15—C14—C13	111.36 (13)	C38—C37—H37A	110.3
C15—C14—H14	124.3	O34—C37—H37B	110.3
C13—C14—H14	124.3	C38—C37—H37B	110.3
C14—C15—S11	113.57 (11)	H37A—C37—H37B	108.5
C14—C15—C115	126.75 (12)	C37—C38—H38A	109.5
S11—C15—C115	119.67 (9)	C37—C38—H38B	109.5
C36—C31—C32	117.42 (13)	H38A—C38—H38B	109.5
C36—C31—C3	122.39 (13)	C37—C38—H38C	109.5
C32—C31—C3	120.20 (12)	H38A—C38—H38C	109.5
C33—C32—C31	121.70 (13)	H38B—C38—H38C	109.5
C33—C32—H32	119.2		

O1—C1—C2—C3	0.0 (3)	C2—C3—C31—C36	4.0 (3)
C12—C1—C2—C3	179.55 (15)	C2—C3—C31—C32	−176.64 (16)
C1—C2—C3—C31	−179.46 (15)	C36—C31—C32—C33	0.5 (2)
O1—C1—C12—C13	−177.31 (17)	C3—C31—C32—C33	−178.92 (15)
C2—C1—C12—C13	3.1 (3)	C31—C32—C33—C34	0.2 (3)
O1—C1—C12—S11	1.2 (2)	C32—C33—C34—O34	179.12 (15)
C2—C1—C12—S11	−178.36 (12)	C32—C33—C34—C35	−0.7 (2)
C15—S11—C12—C13	0.49 (13)	O34—C34—C35—C36	−179.38 (15)
C15—S11—C12—C1	−178.32 (13)	C33—C34—C35—C36	0.5 (2)
C1—C12—C13—C14	178.12 (16)	C34—C35—C36—C31	0.3 (2)
S11—C12—C13—C14	−0.50 (18)	C32—C31—C36—C35	−0.8 (2)
C12—C13—C14—C15	0.2 (2)	C3—C31—C36—C35	178.63 (15)
C13—C14—C15—S11	0.15 (19)	C33—C34—O34—C37	−175.99 (15)
C13—C14—C15—C115	−179.02 (12)	C35—C34—O34—C37	3.9 (2)
C12—S11—C15—C14	−0.37 (14)	C34—O34—C37—C38	169.60 (16)
C12—S11—C15—C115	178.87 (11)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C36—H36 \cdots O1 ⁱ	0.95	2.52	3.4649 (18)	173

Symmetry code: (i) *x*, $-\gamma+3/2$, *z*+1/2.**(IV) (2*E*)-1-(5-Bromothiophen-2-yl)-3-(4-ethoxyphenyl)prop-2-en-1-one***Crystal data*C₁₅H₁₃BrO₂S*M_r* = 337.21Monoclinic, *P*2₁/*c**a* = 16.5498 (7) Å*b* = 7.5069 (4) Å*c* = 11.1574 (5) Å β = 92.618 (4)°*V* = 1384.72 (11) Å³*Z* = 4*F*(000) = 680*D_x* = 1.617 Mg m^{−3}Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 4590 reflections

 θ = 3.3–32.8° μ = 3.11 mm^{−1}*T* = 173 K

Needle, colourless

0.58 × 0.32 × 0.29 mm

*Data collection*Agilent Eos Gemini
diffractometer

Radiation source: Enhance (Mo) X-ray Source

 ω scansAbsorption correction: multi-scan
(CrysAlis RED; Agilent, 2012)*T*_{min} = 0.261, *T*_{max} = 0.405

8866 measured reflections

4040 independent reflections

3189 reflections with *I* > 2σ(*I*)*R*_{int} = 0.037 θ_{max} = 30.0°, θ_{min} = 3.3°*h* = −23→23*k* = −10→7*l* = −15→10*Refinement*Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.038*wR*(*F*²) = 0.078*S* = 1.03

4040 reflections

174 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.027P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.002$$

$$\Delta\rho_{\max} = 0.54 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$$

Extinction correction: SHELXL,

$$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.0142 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.47683 (13)	0.8021 (3)	0.37094 (19)	0.0214 (5)
O1	0.46163 (10)	0.8474 (2)	0.26618 (14)	0.0325 (4)
C2	0.41612 (13)	0.7286 (3)	0.44882 (19)	0.0219 (5)
H2	0.4316	0.6977	0.5292	0.026*
C3	0.33987 (13)	0.7040 (3)	0.40968 (19)	0.0198 (4)
H3	0.3274	0.7351	0.3284	0.024*
S11	0.63142 (3)	0.91205 (8)	0.33350 (5)	0.02358 (14)
C12	0.55978 (13)	0.8208 (3)	0.42271 (19)	0.0197 (4)
C13	0.59275 (14)	0.7744 (3)	0.5328 (2)	0.0237 (5)
H313	0.5625	0.7231	0.5943	0.028*
C14	0.67601 (13)	0.8103 (3)	0.5458 (2)	0.0227 (5)
H14	0.7083	0.7865	0.6165	0.027*
C15	0.70441 (13)	0.8830 (3)	0.44500 (19)	0.0197 (4)
Br15	0.81072 (2)	0.95080 (3)	0.41891 (2)	0.02718 (9)
C31	0.27341 (12)	0.6353 (3)	0.47671 (18)	0.0174 (4)
C32	0.19658 (13)	0.6155 (3)	0.42083 (19)	0.0220 (5)
H32	0.1888	0.6455	0.3384	0.026*
C33	0.13207 (14)	0.5537 (3)	0.4821 (2)	0.0228 (5)
H33	0.0806	0.5404	0.4420	0.027*
C34	0.14246 (13)	0.5107 (3)	0.6032 (2)	0.0201 (4)
C35	0.21844 (14)	0.5284 (3)	0.6608 (2)	0.0214 (5)
H35	0.2259	0.4987	0.7433	0.026*
C36	0.28283 (13)	0.5889 (3)	0.59792 (19)	0.0195 (4)
H36	0.3346	0.5993	0.6378	0.023*
O34	0.07528 (9)	0.4517 (2)	0.65781 (15)	0.0260 (4)
C37	0.08296 (15)	0.4163 (3)	0.7841 (2)	0.0294 (5)
H37A	0.1074	0.5198	0.8270	0.035*
H37B	0.1180	0.3111	0.7997	0.035*
C38	−0.00045 (16)	0.3818 (4)	0.8261 (3)	0.0414 (7)
H38A	−0.0259	0.2861	0.7780	0.062*
H38B	−0.0330	0.4904	0.8172	0.062*
H38C	0.0031	0.3462	0.9107	0.062*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0219 (11)	0.0234 (11)	0.0191 (11)	−0.0007 (9)	0.0017 (8)	−0.0020 (9)
O1	0.0238 (9)	0.0546 (11)	0.0189 (8)	−0.0044 (8)	−0.0014 (7)	0.0074 (8)
C2	0.0217 (11)	0.0276 (12)	0.0164 (11)	−0.0005 (9)	0.0019 (8)	0.0022 (9)
C3	0.0230 (11)	0.0200 (10)	0.0164 (10)	0.0005 (9)	0.0033 (8)	−0.0013 (9)
S11	0.0220 (3)	0.0332 (3)	0.0157 (3)	−0.0040 (2)	0.0026 (2)	0.0027 (2)
C12	0.0191 (11)	0.0228 (11)	0.0173 (10)	−0.0009 (9)	0.0041 (8)	−0.0003 (9)
C13	0.0237 (12)	0.0246 (11)	0.0228 (12)	−0.0026 (10)	0.0019 (9)	0.0039 (10)
C14	0.0225 (11)	0.0236 (11)	0.0216 (11)	−0.0020 (9)	−0.0033 (9)	0.0020 (9)
C15	0.0169 (10)	0.0189 (10)	0.0233 (11)	0.0011 (9)	0.0015 (8)	−0.0016 (9)
Br15	0.01970 (13)	0.02854 (14)	0.03363 (15)	−0.00331 (9)	0.00473 (9)	−0.00198 (10)
C31	0.0179 (10)	0.0166 (10)	0.0176 (10)	0.0017 (8)	0.0012 (8)	−0.0029 (8)
C32	0.0235 (12)	0.0252 (11)	0.0170 (11)	0.0002 (10)	−0.0024 (9)	−0.0011 (9)
C33	0.0162 (11)	0.0269 (12)	0.0250 (12)	−0.0015 (9)	−0.0024 (9)	−0.0018 (10)
C34	0.0177 (11)	0.0195 (10)	0.0232 (11)	0.0006 (9)	0.0030 (9)	−0.0028 (9)
C35	0.0224 (11)	0.0247 (11)	0.0170 (11)	0.0004 (9)	0.0006 (8)	0.0014 (9)
C36	0.0162 (10)	0.0229 (11)	0.0194 (11)	−0.0002 (9)	−0.0009 (8)	−0.0013 (9)
O34	0.0185 (8)	0.0340 (9)	0.0259 (9)	−0.0048 (7)	0.0041 (6)	0.0010 (7)
C37	0.0293 (13)	0.0333 (13)	0.0261 (12)	−0.0032 (11)	0.0074 (10)	0.0028 (11)
C38	0.0353 (15)	0.0489 (16)	0.0414 (16)	−0.0083 (13)	0.0154 (12)	0.0042 (14)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.232 (3)	C32—C33	1.375 (3)
C1—C2	1.466 (3)	C32—H32	0.9500
C1—C12	1.472 (3)	C33—C34	1.392 (3)
C2—C3	1.329 (3)	C33—H33	0.9500
C2—H2	0.9500	C34—O34	1.366 (3)
C3—C31	1.453 (3)	C34—C35	1.392 (3)
C3—H3	0.9500	C35—C36	1.379 (3)
S11—C15	1.708 (2)	C35—H35	0.9500
S11—C12	1.724 (2)	C36—H36	0.9500
C12—C13	1.366 (3)	O34—C37	1.434 (3)
C13—C14	1.405 (3)	C37—C38	1.501 (3)
C13—H313	0.9500	C37—H37A	0.9900
C14—C15	1.353 (3)	C37—H37B	0.9900
C14—H14	0.9500	C38—H38A	0.9800
C15—Br15	1.867 (2)	C38—H38B	0.9800
C31—C36	1.398 (3)	C38—H38C	0.9800
C31—C32	1.398 (3)		
O1—C1—C2	123.4 (2)	C31—C32—H32	119.2
O1—C1—C12	119.5 (2)	C32—C33—C34	119.8 (2)
C2—C1—C12	117.09 (19)	C32—C33—H33	120.1
C3—C2—C1	121.6 (2)	C34—C33—H33	120.1
C3—C2—H2	119.2	O34—C34—C33	116.2 (2)

C1—C2—H2	119.2	O34—C34—C35	124.2 (2)
C2—C3—C31	127.6 (2)	C33—C34—C35	119.7 (2)
C2—C3—H3	116.2	C36—C35—C34	120.0 (2)
C31—C3—H3	116.2	C36—C35—H35	120.0
C15—S11—C12	90.62 (10)	C34—C35—H35	120.0
C13—C12—C1	131.0 (2)	C35—C36—C31	121.3 (2)
C13—C12—S11	111.31 (17)	C35—C36—H36	119.4
C1—C12—S11	117.70 (16)	C31—C36—H36	119.4
C12—C13—C14	113.2 (2)	C34—O34—C37	117.45 (18)
C12—C13—H313	123.4	O34—C37—C38	107.3 (2)
C14—C13—H313	123.4	O34—C37—H37A	110.2
C15—C14—C13	111.6 (2)	C38—C37—H37A	110.2
C15—C14—H14	124.2	O34—C37—H37B	110.2
C13—C14—H14	124.2	C38—C37—H37B	110.2
C14—C15—S11	113.31 (16)	H37A—C37—H37B	108.5
C14—C15—Br15	127.23 (17)	C37—C38—H38A	109.5
S11—C15—Br15	119.46 (12)	C37—C38—H38B	109.5
C36—C31—C32	117.6 (2)	H38A—C38—H38B	109.5
C36—C31—C3	122.2 (2)	C37—C38—H38C	109.5
C32—C31—C3	120.17 (19)	H38A—C38—H38C	109.5
C33—C32—C31	121.7 (2)	H38B—C38—H38C	109.5
C33—C32—H32	119.2		
O1—C1—C2—C3	0.8 (4)	C2—C3—C31—C36	1.7 (4)
C12—C1—C2—C3	−178.9 (2)	C2—C3—C31—C32	−179.1 (2)
C1—C2—C3—C31	−179.0 (2)	C36—C31—C32—C33	0.4 (3)
O1—C1—C12—C13	−176.9 (2)	C3—C31—C32—C33	−178.9 (2)
C2—C1—C12—C13	2.9 (4)	C31—C32—C33—C34	0.6 (3)
O1—C1—C12—S11	1.8 (3)	C32—C33—C34—O34	179.5 (2)
C2—C1—C12—S11	−178.48 (16)	C32—C33—C34—C35	−0.9 (3)
C15—S11—C12—C13	0.85 (18)	O34—C34—C35—C36	179.9 (2)
C15—S11—C12—C1	−178.06 (17)	C33—C34—C35—C36	0.3 (3)
C1—C12—C13—C14	178.1 (2)	C34—C35—C36—C31	0.6 (3)
S11—C12—C13—C14	−0.6 (3)	C32—C31—C36—C35	−1.0 (3)
C12—C13—C14—C15	−0.1 (3)	C3—C31—C36—C35	178.3 (2)
C13—C14—C15—S11	0.8 (3)	C33—C34—O34—C37	−176.45 (19)
C13—C14—C15—Br15	−179.32 (16)	C35—C34—O34—C37	4.0 (3)
C12—S11—C15—C14	−0.92 (18)	C34—O34—C37—C38	171.3 (2)
C12—S11—C15—Br15	179.15 (13)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C36—H36 \cdots O1 ⁱ	0.95	2.52	3.464 (2)	172

Symmetry code: (i) $x, -y+3/2, z+1/2$.

(V) (2E)-3-(4-Bromophenyl)-1-(5-chlorothiophen-2-yl)prop-2-en-1-one

Crystal data

C₁₃H₈BrClOS $M_r = 327.60$ Triclinic, $P\bar{1}$ $a = 6.0152(8) \text{ \AA}$ $b = 8.5691(12) \text{ \AA}$ $c = 13.1824(9) \text{ \AA}$ $\alpha = 75.25(1)^\circ$ $\beta = 81.446(8)^\circ$ $\gamma = 70.281(12)^\circ$ $V = 617.09(14) \text{ \AA}^3$ $Z = 2$ $F(000) = 324$ $D_x = 1.763 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4058 reflections

 $\theta = 3.2\text{--}32.8^\circ$ $\mu = 3.69 \text{ mm}^{-1}$ $T = 173 \text{ K}$

Needle, colourless

 $0.41 \times 0.20 \times 0.18 \text{ mm}$

Data collection

Agilent Eos Gemini
diffractometer

Radiation source: Enhance (Mo) X-ray Source

 ω scansAbsorption correction: multi-scan
(CrysAlis RED; Agilent, 2012) $T_{\min} = 0.298$, $T_{\max} = 0.514$

6674 measured reflections

3599 independent reflections

2817 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.026$ $\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.2^\circ$ $h = -8 \rightarrow 8$ $k = -10 \rightarrow 12$ $l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.100$ $S = 1.03$

3599 reflections

154 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.3839P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 1.43 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.53 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3079 (5)	0.7031 (4)	0.5481 (2)	0.0219 (5)
O1	0.0931 (3)	0.7382 (3)	0.54196 (16)	0.0316 (5)
C2	0.4552 (5)	0.7932 (4)	0.4740 (2)	0.0233 (5)
H2	0.6221	0.7524	0.4781	0.028*
C3	0.3594 (5)	0.9316 (4)	0.4008 (2)	0.0235 (5)
H3	0.1914	0.9731	0.4024	0.028*
S11	0.25627 (12)	0.46020 (9)	0.72271 (5)	0.02442 (15)
C12	0.4287 (5)	0.5637 (3)	0.63199 (19)	0.0209 (5)
C13	0.6614 (5)	0.4996 (4)	0.6548 (2)	0.0230 (5)
H13	0.7821	0.5412	0.6141	0.028*

C14	0.7041 (5)	0.3649 (4)	0.7453 (2)	0.0234 (5)
H14	0.8555	0.3055	0.7719	0.028*
C15	0.5014 (5)	0.3316 (3)	0.7890 (2)	0.0228 (5)
Cl15	0.47267 (14)	0.18017 (10)	0.89894 (5)	0.03458 (18)
C31	0.4883 (5)	1.0255 (3)	0.3183 (2)	0.0215 (5)
C32	0.3621 (5)	1.1691 (3)	0.2476 (2)	0.0236 (5)
H32	0.1943	1.2092	0.2575	0.028*
C33	0.4754 (5)	1.2549 (4)	0.1632 (2)	0.0250 (5)
H33	0.3868	1.3509	0.1151	0.030*
C34	0.7182 (5)	1.1974 (4)	0.1513 (2)	0.0247 (6)
Br34	0.87986 (6)	1.31203 (4)	0.03812 (2)	0.03776 (12)
C35	0.8519 (5)	1.0562 (4)	0.2200 (2)	0.0268 (6)
H35	1.0197	1.0177	0.2099	0.032*
C36	0.7356 (5)	0.9725 (4)	0.3037 (2)	0.0255 (6)
H36	0.8255	0.8775	0.3519	0.031*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0238 (13)	0.0213 (13)	0.0195 (12)	−0.0059 (10)	−0.0023 (10)	−0.0036 (10)
O1	0.0206 (10)	0.0337 (12)	0.0348 (11)	−0.0058 (9)	−0.0044 (8)	0.0004 (9)
C2	0.0221 (12)	0.0250 (14)	0.0221 (12)	−0.0089 (11)	−0.0016 (10)	−0.0022 (10)
C3	0.0238 (13)	0.0227 (14)	0.0232 (12)	−0.0067 (11)	−0.0019 (10)	−0.0043 (10)
S11	0.0193 (3)	0.0278 (4)	0.0247 (3)	−0.0094 (3)	0.0003 (2)	−0.0015 (3)
C12	0.0232 (13)	0.0203 (13)	0.0196 (11)	−0.0090 (10)	−0.0011 (10)	−0.0023 (9)
C13	0.0223 (12)	0.0255 (14)	0.0226 (12)	−0.0108 (11)	−0.0015 (10)	−0.0031 (10)
C14	0.0216 (12)	0.0241 (14)	0.0231 (12)	−0.0054 (11)	−0.0050 (10)	−0.0029 (10)
C15	0.0259 (13)	0.0216 (13)	0.0206 (12)	−0.0084 (11)	−0.0022 (10)	−0.0025 (10)
Cl15	0.0439 (4)	0.0324 (4)	0.0251 (3)	−0.0175 (3)	−0.0011 (3)	0.0046 (3)
C31	0.0228 (13)	0.0213 (13)	0.0206 (12)	−0.0070 (10)	−0.0027 (10)	−0.0043 (10)
C32	0.0227 (13)	0.0203 (14)	0.0237 (12)	−0.0013 (10)	−0.0050 (10)	−0.0035 (10)
C33	0.0279 (14)	0.0201 (13)	0.0242 (13)	−0.0045 (11)	−0.0082 (11)	−0.0005 (10)
C34	0.0307 (14)	0.0243 (14)	0.0206 (12)	−0.0133 (12)	−0.0031 (10)	−0.0005 (10)
Br34	0.03904 (19)	0.0438 (2)	0.02867 (17)	−0.02171 (15)	−0.00236 (12)	0.00715 (13)
C35	0.0224 (13)	0.0262 (15)	0.0302 (14)	−0.0080 (11)	−0.0060 (11)	−0.0005 (11)
C36	0.0217 (13)	0.0247 (14)	0.0259 (13)	−0.0054 (11)	−0.0061 (10)	0.0018 (10)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.234 (3)	C14—H14	0.9500
C1—C12	1.469 (4)	C15—Cl15	1.712 (3)
C1—C2	1.469 (4)	C31—C32	1.398 (4)
C2—C3	1.338 (4)	C31—C36	1.400 (4)
C2—H2	0.9500	C32—C33	1.393 (4)
C3—C31	1.469 (4)	C32—H32	0.9500
C3—H3	0.9500	C33—C34	1.374 (4)
S11—C15	1.718 (3)	C33—H33	0.9500
S11—C12	1.733 (3)	C34—C35	1.390 (4)

C12—C13	1.369 (4)	C34—Br34	1.898 (3)
C13—C14	1.418 (4)	C35—C36	1.388 (4)
C13—H13	0.9500	C35—H35	0.9500
C14—C15	1.357 (4)	C36—H36	0.9500
O1—C1—C12	120.0 (2)	C14—C15—S11	113.4 (2)
O1—C1—C2	123.2 (2)	Cl15—C15—S11	119.76 (16)
C12—C1—C2	116.8 (2)	C32—C31—C36	117.8 (2)
C3—C2—C1	121.4 (2)	C32—C31—C3	119.6 (2)
C3—C2—H2	119.3	C36—C31—C3	122.6 (2)
C1—C2—H2	119.3	C33—C32—C31	121.8 (3)
C2—C3—C31	126.4 (3)	C33—C32—H32	119.1
C2—C3—H3	116.8	C31—C32—H32	119.1
C31—C3—H3	116.8	C34—C33—C32	118.4 (2)
C15—S11—C12	90.62 (13)	C34—C33—H33	120.8
C13—C12—C1	131.2 (2)	C32—C33—H33	120.8
C13—C12—S11	111.4 (2)	C33—C34—C35	122.0 (2)
C1—C12—S11	117.45 (19)	C33—C34—Br34	119.8 (2)
C12—C13—C14	113.2 (2)	C35—C34—Br34	118.3 (2)
C12—C13—H13	123.4	C36—C35—C34	118.7 (3)
C14—C13—H13	123.4	C36—C35—H35	120.6
C15—C14—C13	111.4 (2)	C34—C35—H35	120.6
C15—C14—H14	124.3	C35—C36—C31	121.3 (3)
C13—C14—H14	124.3	C35—C36—H36	119.4
C14—C15—Cl15	126.9 (2)	C31—C36—H36	119.4
O1—C1—C2—C3	6.9 (4)	C12—S11—C15—C14	0.1 (2)
C12—C1—C2—C3	−173.8 (3)	C12—S11—C15—Cl15	−179.89 (18)
C1—C2—C3—C31	−175.7 (2)	C2—C3—C31—C32	−179.7 (3)
O1—C1—C12—C13	179.2 (3)	C2—C3—C31—C36	3.6 (4)
C2—C1—C12—C13	−0.1 (4)	C36—C31—C32—C33	1.9 (4)
O1—C1—C12—S11	−2.7 (4)	C3—C31—C32—C33	−175.0 (2)
C2—C1—C12—S11	177.99 (19)	C31—C32—C33—C34	−1.3 (4)
C15—S11—C12—C13	−0.4 (2)	C32—C33—C34—C35	0.7 (4)
C15—S11—C12—C1	−178.8 (2)	C32—C33—C34—Br34	−179.3 (2)
C1—C12—C13—C14	178.7 (3)	C33—C34—C35—C36	−0.7 (4)
S11—C12—C13—C14	0.6 (3)	Br34—C34—C35—C36	179.3 (2)
C12—C13—C14—C15	−0.5 (3)	C34—C35—C36—C31	1.4 (4)
C13—C14—C15—Cl15	−179.8 (2)	C32—C31—C36—C35	−2.0 (4)
C13—C14—C15—S11	0.1 (3)	C3—C31—C36—C35	174.9 (3)

(VI) (2E)-1-(5-Bromothiophen-2-yl)-3-(3-methoxyphenyl)prop-2-en-1-one

Crystal data

C₁₄H₁₁BrO₂S*M_r* = 323.19Monoclinic, *P*2₁/*c**a* = 9.2726 (6) Å*b* = 11.3948 (8) Å*c* = 12.1472 (7) Å β = 93.273 (6)°*V* = 1281.37 (14) Å³

$Z = 4$
 $F(000) = 648$
 $D_x = 1.675 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 4224 reflections

$\theta = 3.4\text{--}32.7^\circ$
 $\mu = 3.36 \text{ mm}^{-1}$
 $T = 173 \text{ K}$
 Block, colourless
 $0.54 \times 0.42 \times 0.31 \text{ mm}$

Data collection

Agilent Eos Gemini
 diffractometer
 Radiation source: Enhance (Mo) X-ray Source
 ω scans
 Absorption correction: multi-scan
 (CrysAlis RED; Agilent, 2012)
 $T_{\min} = 0.216$, $T_{\max} = 0.353$
 8260 measured reflections

3722 independent reflections
 2914 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.4^\circ$
 $h = -13 \rightarrow 12$
 $k = -15 \rightarrow 16$
 $l = -16 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.075$
 $S = 1.02$
 3722 reflections
 164 parameters
 0 restraints

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0293P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.003$
 $\Delta\rho_{\max} = 0.49 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.46 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4180 (2)	0.29186 (18)	0.72203 (17)	0.0205 (4)
O1	0.41175 (17)	0.39954 (13)	0.71617 (13)	0.0310 (4)
C2	0.5005 (2)	0.22053 (19)	0.64696 (17)	0.0217 (4)
H2	0.5078	0.1382	0.6584	0.026*
C3	0.5653 (2)	0.26916 (19)	0.56325 (15)	0.0204 (4)
H3	0.5545	0.3516	0.5549	0.024*
S11	0.22557 (6)	0.31118 (4)	0.88317 (4)	0.02124 (12)
C12	0.3390 (2)	0.23001 (18)	0.80567 (16)	0.0180 (4)
C13	0.3392 (2)	0.11390 (18)	0.83642 (17)	0.0232 (4)
H13	0.3952	0.0553	0.8031	0.028*
C14	0.2474 (2)	0.09085 (19)	0.92288 (17)	0.0245 (5)
H14	0.2342	0.0155	0.9542	0.029*
C15	0.1805 (2)	0.18936 (17)	0.95556 (16)	0.0192 (4)
Br15	0.05413 (2)	0.20408 (2)	1.06918 (2)	0.02654 (8)
C31	0.6513 (2)	0.20960 (18)	0.48261 (16)	0.0178 (4)
C32	0.7142 (2)	0.27779 (18)	0.40207 (16)	0.0185 (4)
H32	0.6980	0.3601	0.3996	0.022*

C33	0.7994 (2)	0.22577 (18)	0.32643 (16)	0.0192 (4)
C34	0.8214 (2)	0.10536 (18)	0.32844 (17)	0.0242 (5)
H34	0.8796	0.0695	0.2761	0.029*
C35	0.7583 (2)	0.0380 (2)	0.40685 (17)	0.0279 (5)
H35	0.7730	−0.0445	0.4078	0.034*
C36	0.6737 (2)	0.08920 (18)	0.48427 (17)	0.0237 (5)
H36	0.6314	0.0420	0.5382	0.028*
O33	0.86717 (16)	0.28429 (13)	0.24569 (13)	0.0256 (3)
C37	0.8641 (3)	0.40869 (19)	0.24766 (19)	0.0309 (5)
H37A	0.9166	0.4394	0.1861	0.046*
H37B	0.9099	0.4368	0.3174	0.046*
H37C	0.7637	0.4358	0.2408	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0191 (10)	0.0234 (11)	0.0194 (10)	0.0018 (8)	0.0043 (8)	−0.0007 (9)
O1	0.0368 (9)	0.0211 (8)	0.0371 (9)	0.0005 (7)	0.0201 (7)	0.0008 (7)
C2	0.0216 (10)	0.0232 (10)	0.0208 (10)	0.0029 (9)	0.0072 (8)	−0.0020 (9)
C3	0.0200 (10)	0.0211 (10)	0.0204 (10)	0.0034 (8)	0.0038 (8)	−0.0012 (9)
S11	0.0244 (3)	0.0195 (2)	0.0210 (3)	0.0029 (2)	0.0104 (2)	−0.0005 (2)
C12	0.0177 (10)	0.0208 (10)	0.0161 (9)	0.0013 (8)	0.0051 (7)	−0.0045 (8)
C13	0.0277 (11)	0.0197 (10)	0.0229 (11)	0.0033 (9)	0.0071 (8)	−0.0024 (9)
C14	0.0305 (12)	0.0203 (10)	0.0237 (11)	0.0000 (9)	0.0099 (9)	0.0023 (9)
C15	0.0179 (10)	0.0249 (11)	0.0152 (9)	−0.0025 (8)	0.0042 (7)	0.0017 (8)
Br15	0.02519 (12)	0.03558 (14)	0.01992 (12)	0.00135 (9)	0.01063 (8)	0.00304 (9)
C31	0.0153 (9)	0.0249 (10)	0.0133 (9)	−0.0004 (8)	0.0020 (7)	−0.0016 (8)
C32	0.0194 (10)	0.0184 (9)	0.0179 (9)	0.0011 (8)	0.0026 (7)	−0.0005 (8)
C33	0.0175 (10)	0.0233 (10)	0.0169 (9)	0.0017 (8)	0.0029 (7)	0.0029 (9)
C34	0.0296 (11)	0.0234 (10)	0.0206 (10)	0.0044 (9)	0.0100 (8)	−0.0038 (9)
C35	0.0384 (13)	0.0199 (10)	0.0265 (11)	0.0052 (9)	0.0103 (9)	0.0017 (9)
C36	0.0306 (11)	0.0214 (11)	0.0197 (10)	−0.0008 (9)	0.0079 (8)	0.0025 (9)
O33	0.0301 (8)	0.0232 (8)	0.0252 (8)	0.0013 (6)	0.0166 (6)	0.0030 (7)
C37	0.0375 (13)	0.0220 (11)	0.0346 (13)	−0.0010 (10)	0.0147 (10)	0.0066 (10)

Geometric parameters (Å, °)

C1—O1	1.230 (2)	C31—C36	1.388 (3)
C1—C12	1.467 (3)	C31—C32	1.402 (3)
C1—C2	1.469 (3)	C32—C33	1.380 (3)
C2—C3	1.331 (3)	C32—H32	0.9500
C2—H2	0.9500	C33—O33	1.368 (2)
C3—C31	1.465 (3)	C33—C34	1.387 (3)
C3—H3	0.9500	C34—C35	1.379 (3)
S11—C15	1.708 (2)	C34—H34	0.9500
S11—C12	1.7206 (19)	C35—C36	1.387 (3)
C12—C13	1.375 (3)	C35—H35	0.9500
C13—C14	1.414 (3)	C36—H36	0.9500

C13—H13	0.9500	O33—C37	1.418 (2)
C14—C15	1.353 (3)	C37—H37A	0.9800
C14—H14	0.9500	C37—H37B	0.9800
C15—Br15	1.8681 (18)	C37—H37C	0.9800
O1—C1—C12	119.71 (18)	C32—C31—C3	118.29 (19)
O1—C1—C2	122.72 (19)	C33—C32—C31	120.24 (19)
C12—C1—C2	117.57 (19)	C33—C32—H32	119.9
C3—C2—C1	121.2 (2)	C31—C32—H32	119.9
C3—C2—H2	119.4	O33—C33—C32	124.89 (19)
C1—C2—H2	119.4	O33—C33—C34	114.96 (17)
C2—C3—C31	127.2 (2)	C32—C33—C34	120.15 (18)
C2—C3—H3	116.4	C35—C34—C33	119.68 (18)
C31—C3—H3	116.4	C35—C34—H34	120.2
C15—S11—C12	91.15 (10)	C33—C34—H34	120.2
C13—C12—C1	131.11 (18)	C34—C35—C36	120.8 (2)
C13—C12—S11	111.13 (14)	C34—C35—H35	119.6
C1—C12—S11	117.76 (15)	C36—C35—H35	119.6
C12—C13—C14	112.87 (18)	C35—C36—C31	119.76 (19)
C12—C13—H13	123.6	C35—C36—H36	120.1
C14—C13—H13	123.6	C31—C36—H36	120.1
C15—C14—C13	111.69 (18)	C33—O33—C37	117.69 (16)
C15—C14—H14	124.2	O33—C37—H37A	109.5
C13—C14—H14	124.2	O33—C37—H37B	109.5
C14—C15—S11	113.17 (15)	H37A—C37—H37B	109.5
C14—C15—Br15	127.49 (15)	O33—C37—H37C	109.5
S11—C15—Br15	119.32 (11)	H37A—C37—H37C	109.5
C36—C31—C32	119.33 (18)	H37B—C37—H37C	109.5
C36—C31—C3	122.38 (18)		
O1—C1—C2—C3	−4.7 (4)	C12—S11—C15—Br15	178.16 (13)
C12—C1—C2—C3	174.42 (19)	C2—C3—C31—C36	1.0 (3)
C1—C2—C3—C31	179.66 (19)	C2—C3—C31—C32	−178.2 (2)
O1—C1—C12—C13	−172.7 (2)	C36—C31—C32—C33	−1.0 (3)
C2—C1—C12—C13	8.2 (4)	C3—C31—C32—C33	178.14 (18)
O1—C1—C12—S11	6.5 (3)	C31—C32—C33—O33	−179.10 (19)
C2—C1—C12—S11	−172.61 (16)	C31—C32—C33—C34	1.1 (3)
C15—S11—C12—C13	0.11 (17)	O33—C33—C34—C35	179.8 (2)
C15—S11—C12—C1	−179.23 (17)	C32—C33—C34—C35	−0.4 (3)
C1—C12—C13—C14	179.2 (2)	C33—C34—C35—C36	−0.4 (4)
S11—C12—C13—C14	0.0 (2)	C34—C35—C36—C31	0.5 (4)
C12—C13—C14—C15	−0.2 (3)	C32—C31—C36—C35	0.2 (3)
C13—C14—C15—S11	0.3 (3)	C3—C31—C36—C35	−178.9 (2)
C13—C14—C15—Br15	−177.96 (15)	C32—C33—O33—C37	7.9 (3)
C12—S11—C15—C14	−0.22 (18)	C34—C33—O33—C37	−172.3 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C13—H13 \cdots O1 ⁱ	0.95	2.54	3.446 (3)	159

Symmetry code: (i) $-x+1, y-1/2, -z+3/2$.